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IS 10271 (1982): Anhydrous hydrogen fluoride, technical
[CHD 1: Inorganic Chemicals]



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IS : 10271 - 1982

Indian Standard
SPECIFICATION FOR
ANHYDROUS HYDROGEN FLUORIDE,
TECHNICAL

UDC 661.487



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MANAK BHAVAN, 9 BAHADUR SHAH ZAFAR MARG
NEW DELHI 110002

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March 1983

**AMENDMENT NO. 2 JANUARY 2000
TO
IS 10271 : 1982 SPECIFICATION FOR ANHYDROUS
HYDROGEN FLUORIDE, TECHNICAL**

[*Page 4, Table 1, Sl No. (v)*] — Substitute ' P_2O_5 ' for ' P_2O_6 '.

(CHD 2)

Reprography Unit, BIS, New Delhi, India

AMENDMENT NO. 3 JUNE 2005
TO
IS 10271 : 1982 SPECIFICATION FOR
ANHYDROUS HYDROGEN FLUORIDE, TECHNICAL

(Page 10, clause A-5.2.6.2) — Substitute 'sodium' for 'sodiums'.

(Page 12, clause A-6.2.2, line 2) — Substitute '[(NH₄)₆ Mo₇O₂₄, 4H₂O]' for '[(NH₄)₆ Mo₇O₂₄]'.

(Page 12, clause A-6.2.3.1, line 1) — Substitute 'naphol' for 'nepthol'.

(Page 13, clause A-6.2.4.1, line 5) — Substitute 'Transfer' for 'Trasfer'.

(CHD I)

Reprography Unit, BIS, New Delhi, India



AMENDMENT NO. 1 OCTOBER 1984

TO

IS:10271-1982 SPECIFICATION FOR HYDROFLUORIC
ACID, AQUEOUS

Corrigenda

[Page 4, Table 1, Sl No.(v), col 3] - Substitute
'0.07' for '0.7'.

[Page 4, Table 1, Sl No.(vi), col 3] - Substitute
'99.70' for '99.10'.

(Page 6, clause A-2.2.1, line 2) - Substitute
'iodide' for 'iodine' and '12.7' for '12'.

(Page 7, clause A-2.2.4, line 1) - Substitute
'5g/l' for '5g/ml'.

(Page 12, clause A-5.5, calculation, lines 2
and 3) - Substitute 'µg' for 'g'.

(CDC 56)

Indian Standard

SPECIFICATION FOR ANHYDROUS HYDROGEN FLUORIDE, TECHNICAL

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Indian Standard
**SPECIFICATION FOR
ANHYDROUS HYDROGEN FLUORIDE,
TECHNICAL**

0. FOREWORD

0.1 This Indian Standard was adopted by the Indian Standards Institution on 31 August 1982, after the draft finalized by the Acids, Alkalis and Halides Sectional Committee had been approved by the Chemical Division Council.

0.2 For general information regarding precautions to be observed in the safe handling and use of anhydrous hydrogen fluoride, reference may be made to IS:5184-1969*.

0.3 The filling, transport and importation regulations of anhydrous hydrogen fluoride in cylinders are governed by the *Gas Cylinders Rules, 1940*.

0.4 In the preparation of this standard reference has been made to the following publication of International Organization for Standardization (ISO):

ISO 3137-1974 Anhydrous hydrogen fluoride for industrial use — Sampling

ISO 3138-1974 Anhydrous hydrogen fluoride for industrial use — Determination of non-volatile acid content — Titrimetric method

ISO 3699-1976 Anhydrous hydrogen fluoride for industrial use — Determination of water content — Karl Fischer method

ISO 3701-1976 Anhydrous hydrogen fluoride for industrial use — Determination of hexafluoro-silicic acid content — Reduced molybdosilicate photometric method

ISO 3702-1976 Anhydrous hydrogen fluoride for industrial use — Determination of sulphur dioxide content — Iodometric method.

*Code of safety for hydrofluoric acid.

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0.5 For the purpose of deciding whether a particular requirement of this standard is complied with, the final value, observed or calculated, expressing the result of a test or analysis, shall be rounded off in accordance with IS : 2-1960*. The number of significant places retained in the rounded off value should be the same as that of the specified value in this standard.

1. SCOPE

1.1 This standard prescribes the requirements and the methods of sampling and test for anhydrous hydrogen fluoride, technical.

2. REQUIREMENTS

2.1 Description

2.1.1 The material shall be liquid under pressure, free from sediment and other impurities.

2.1.2 The material shall also comply with the requirements laid down in Table 1 when tested in accordance with the methods prescribed in Appendix A. Reference to the relevant clauses of Appendix A is given in col 4 of the table.

TABLE 1 REQUIREMENTS FOR ANHYDROUS HYDROGEN FLUORIDE, TECHNICAL

SL No.	CHARACTERISTIC	REQUIREMENT	METHOD OF TESTS (REF TO CL No. OF APPENDIX A)
(1)	(2)	(3)	(4)
i)	Sulphur dioxide (as SO_2), percent by mass, <i>Max</i>	0.02	A-2
ii)	Moisture, percent by mass, <i>Max</i>	0.08	A-3
iii)	Non-volatile acid (as H_2SO_4), percent by mass, <i>Max</i>	0.05	A-4
iv)	Silica (as H_2SiF_6), percent by mass, <i>Max</i>	0.05	A-5
v)	Phosphorus (as P_2O_5), percent by mass, <i>Max</i>	0.7	A-6
vi)	Hydrogen fluoride (HF) content, percent by mass, <i>Min</i>	99.10	A-7

*Rules for rounding off numerical values (revised).

3. PACKING AND MARKING

3.1 Packing

3.1.1 Anhydrous hydrogen fluoride shall be packed in steel containers equipped with a special valve for discharge. An outage space shall be provided above the liquid level to compensate for liquid expansion as temperature increases. The filling ratio shall be kept 0.85.

3.1.2 The packing shall be further subject to the relevant provisions of *Red Tariff No. 18 of 1960*, issued by the Indian Railways Conference Association, with any alterations and additions made thereunder.

3.2 Marking

3.2.1 The containers and also the packages, where possible, shall be suitably marked in red letters, not less than 25 mm high, showing the following information:

- a) Name and grade (technical) of the material;
- b) Manufacturer's name and/or his recognized trade-mark, if any;
- c) Mass of the material;
- d) Year of manufacture; and
- e) Lot number.

3.2.2 They shall prominently display the following words:

HYDROGEN FLUORIDE ANHYDROUS : DANGER :
EXTREMELY HAZARDOUS. LIQUID AND VAPOUR
CAUSE SEVERE BURNS WHICH MAY NOT BE IMME-
DIATELY PAINFUL OR VISIBLE.

DO NOT GET IN EYES, ON SKIN, ON CLOTHING. DO
NOT BREATHE VAPOUR. STORE OUT OF SUN AND
AWAY FROM DIRECT HEAT.

3.2.3 The packages shall be labelled as shown in Fig. 15 of IS : 1260 (Part I)-1973*.

3.2.4 Unloading of the material shall be done in accordance with the practices recommended as in IS : 5184-1969†.

*Pictorial markings for handling and labelling of goods: Part I Dangerous goods (first revision).

†Code of safety for hydrofluoric acid.

3.2.5 The containers may also be marked with the ISI Certification Mark.

NOTE— The use of the ISI Certification Mark is governed by the provisions of the Indian Standards Institution (Certification Marks) Act and the Rules and Regulations made thereunder. The ISI Mark on products covered by an Indian Standard conveys the assurance that they have been produced to comply with the requirements of that standard under a well-defined system of inspection, testing and quality control which is devised and supervised by ISI and operated by the producer. ISI marked products are also continuously checked by ISI for conformity to that standard as a further safeguard. Details of conditions under which a licence for the use of the ISI Certification Mark may be granted to manufacturers or processors, may be obtained from the Indian Standards Institution.

4. SAMPLING

4.1 The method of drawing representative samples and the criteria for conformity of the material to the specification requirements shall be as prescribed in Appendix B.

A P P E N D I X A

(Clause 2.1.2)

METHODS OF TEST FOR ANHYDROUS HYDROGEN FLUORIDE, TECHNICAL

A-1. QUALITY OF REAGENTS

A-1.1 Unless specified otherwise, pure chemicals and distilled water (*see* IS : 1070-1977*) shall be used in tests.

NOTE— ' Pure chemicals ' shall mean chemicals that do not contain impurities which affect the results of analysis.

A-2. DETERMINATION OF SULPHUR DIOXIDE CONTENT

A-2.1 Principle of Method — Addition of a measured volume of standard iodine solution to a test portion and back titration of the excess iodine with standard sodium thiosulphate solution and calculation of sulphur dioxide content from the volume of iodine used.

A-2.2 Reagents

A-2.2.1 Iodine Solution — (Approximately 0.1 N). Dissolve 20 g of potassium iodine in about 50 ml of water. Add 127 g of iodine and stir to dissolve. Transfer this solution to a 1 000 ml measuring flask and make up the volume to the mark with water and mix thoroughly. Prepare fresh solution at the time of use.

*Specification for water for general laboratory use (*second revision*).

A-2.2.2 Iodine Solution — (Approximately 0.01 N). Take 100 ml of 0.1 N iodine solution in 1 000-ml measuring flask and dilute up to the mark with water and mix thoroughly. Prepare fresh solution at the time of use.

A-2.2.3 Sodium Thiosulphate Solution (0.01 N) — Dissolve 2.482 g of sodium thiosulphate hydrated salt in about 100 ml of water and transfer into a 1 000-ml measuring flask. Make up the volume to the mark with water and mix thoroughly. Standardize with potassium dichromate solution.

A-2.2.4 Starch Indicator Solution (5 g/ml) — Disperse 0.5 g of soluble starch in a few millilitres of cold water and add the dispersion to 100 ml of boiling water. Stir and mix the fresh solution before use.

A-2.3 Apparatus

A-2.3.1 Polyethylene Weighing Bottle — 100-ml capacity.

A-2.3.2 Polyethylene Beaker — 500-ml capacity.

A-2.3.3 Polyethylene Stirrer Rod

A-2.3.4 Burette — 25×0.1 ml capacity, whole pipette (bulb pipette) 50-ml capacity.

A-2.4 Procedure

A-2.4.1 Transfer about 25 ml of the sample prepared as specified in B-5.3 into the preweighed weighing bottle, replace the lid and weigh again to the nearest 0.1 g.

A-2.4.2 Place 100 ml of water in the 500-ml beaker, add 50 ml of 0.01 N iodine solution and transfer the test sample, weighed as above, quantitatively to the mixture. Allow to stand for a few minutes and titrate excess of iodine with standard sodium thiosulphate solution until the solution becomes pale yellow in colour. Add 2 ml of starch indicator which turns the solutions blue and continue the titration until the solution is colourless; use polyethylene rod for stirring.

A-2.4.3 Carry out the standardization of the iodine solution, by the same method as mentioned above in case of test sample using 100 ml of water and 50 ml of the iodine solution, but omitting the test portion.

A-2.5 Calculation

$$\text{Sulphur dioxide (as SO}_2 \text{)} \\ \text{percent by mass} = \frac{3.2 (V_1 - V_2)}{M \times C}$$

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where

V_1 = volume in ml of the standard sodium thiosulphate solution used in standardization of 0.01 N iodine solution,

V_2 = volume in ml of the standard sodium thiosulphate solution used in the test,

M = mass in g of the test portion,

C = concentration of anhydrous hydrogen fluoride expressed as percent by mass in the test sample (*see B-5.4*), and 3.2 is factor giving mass in g of sulphur dioxide (as SO_2), corresponding to 1 ml of 0.01 N sodium thiosulphate solution.

A-3. DETERMINATION OF WATER CONTENT

A-3.1 Procedure — Determine water by Karl Fischer method prescribed in IS : 2362-1973*.

A-4. DETERMINATION OF NON-VOLATILE ACID CONTENT (SULPHURIC ACID AND FLUOSULPHONIC ACID)

A-4.1 Principle of the Method — Elimination of the volatile acid by evaporation and titration of the rest non-volatile acid with sodium hydroxide solution in presence of phenolphthalein indicator.

A-4.2 Reagents

A-4.2.1 Sodium Hydroxide Standard Solution — 0.01 N.

A-4.2.2 Phenolphthalein Indicator Solution — Dissolve 1 g of phenolphthalein powder in 100 ml of ethanol (95 percent *v/v*). Make the solution neutral by adding slowly a few drops of sodium hydroxide solution till pale rose colour is achieved.

A-4.3 Apparatus

A-4.3.1 Platinum Dish — 100-ml capacity.

A-4.4 Procedure

A-4.4.1 Weigh 50 g of the diluted sample prepared as specified in **B-5.3** in the platinum dish and keep the dish on water bath for evaporation. Evaporate nearly to dryness.

*Determination of water by Karl Fischer method (*first revision*).

A-4.4.2 Add 50 ml of water and evaporate again to dryness. Repeat the process, adding water and evaporating the mass to dryness two times more so as to remove hydrogen fluoride gas completely.

A-4.4.3 Add 50 ml of water and transfer the contents quantitatively to a 250-ml glass beaker. Add 1 g of neutral sodium fluoride powder to inhibit any aluminium or iron present, then 2 to 3 drops of phenolphthalein indicator solution and titrate with sodium hydroxide solution till pale pink colouration persists.

A-4.5 Calculation

$$\begin{array}{l} \text{Non-volatile acids (as H}_2\text{SO}_4 \text{),} \\ \text{percent by mass} \end{array} = \frac{V \times 4.9}{M \times C}$$

where

V = volume in ml of sodium hydroxide solution consumed in titration,

M = mass in g of the test portion,

C = concentration of anhydrous hydrogen fluoride expressed as percentage by mass in the test sample, and

4.9 is factor giving mass in g of sulphuric acid corresponding to 1 ml of 0.01 N sodium hydroxide solution.

A-5. DETERMINATION OF SILICA AS HYDROFLUOSILICIC ACID

A-5.1 Principle of the Method

A-5.1.1 Formation of molybdosilicate complex (yellow) in weakly acidic medium in presence of boric acid (to suppress fluorine interference). Reduction of the yellow complex, after addition of sulphuric acid and oxalic acid (to eliminate phosphate interference). Photometric measurement of the intensity of blue colour complex formed at 580 nm wave length.

A-5.2 Reagents

A-5.2.1 *Dilute Sulphuric Acid* — approximately 9 N.

A-5.2.2 *Dilute Hydrochloric Acid* — approximately 2 N.

A-5.2.3 *Boric Acid Solution* — 40 g/l.

A-5.2.4 *Oxalic Acid Solution* — 100 g anhydrous acid per litre.

A-5.2.5 Ammonium Molybdate Solution — 100 g/l.

A-5.2.5.1 Dissolve 25 g of ammonium molybdate tetrahydrate $[(\text{NH}_4)_6\text{Mo}_7\text{O}_{24} \cdot 4\text{H}_2\text{O}]$ in 200 ml of water at about 50 to 60°C. Allow to cool to ambient temperature. Filter, if necessary. Transfer to a 250-ml measuring flask and dilute to the mark with water. Mix thoroughly and transfer immediately to a polyethylene bottle.

A-5.2.5.2 Discard the solution when precipitates are formed.

A-5.2.6 Reducing Solution

A-5.2.6.1 Dissolve 7 g of anhydrous sodium sulphite (Na_2SO_3) in about 50 ml of water. Add 1.5 g of 4-amino-3-hydroxynaphthalene-1-sulphonic acid (also known as 1-amino-2-naphthol-4-sulphonic acid) and dissolve by stirring.

A-5.2.6.2 Dissolve 90 g of sodiums metabisulphite ($\text{Na}_2\text{S}_2\text{O}_5$) in 900 ml of water.

A-5.2.6.3 Mix both the above solutions and dilute to 1 000 ml. Filter, if necessary and store in a cool dark place.

A-5.2.7 Silica Standard Solutions

A-5.2.7.1 Standard solution A — (corresponding to 1 000 ppm of hexafluorosilicic acid). Weigh accurately 0.417 g of pure quartz finely ground and dried powder in a 25-ml platinum crucible, add 5 g of anhydrous sodium carbonate and mix thoroughly with a thick platinum wire. Fuse the mixture gently till a clear solution is obtained.

Allow to cool, add warm water and heat moderately till the silicate dissolves completely. Cool, transfer quantitatively to 1 000-ml measuring flask, dilute to the mark and mix. Transfer immediately to a polyethylene bottle. One millilitre of this solution contains the equivalent of 1.00 mg of hydrofluorosilicic acid.

A-5.2.7.2 Standard solution B — (corresponding to 20 ppm of hydrofluosilicic acid). Transfer 20.0 ml of standard solution A into a 1 000-ml measuring flask, dilute to the mark and mix. Transfer to a polyethylene bottle. One millilitre of this solution contains the equivalent of 20 micro gram. Prepare this solution at the time of use.

NOTE—Store all these reagents in polyethylene containers and carry out all operations in apparatus of this material except where specified otherwise.

A-5.3 Apparatus

A-5.3.1 Platinum Crucible — 25 ml capacity and platinum rod.

A-5.3.2 Photoelectric Absorptiometer — fitted with cells of 2 cm optical path and with filters allowing maximum transmission at about 610 nm wave length.

A-5.4 Procedure

A-5.4.1 Introduce a quantity of test solution prepared by the method specified in **B-5.3**, containing not more than 0.5 g of hydrogen fluoride into a tared 100-ml polyethylene beaker and weigh again to the nearest 0.001 g. Dilute to about 20 ml with water and add while stirring 4 ml of dilute hydrochloric acid and 5 ml of boric acid solution.

A-5.4.2 Allow to stand for 5 minutes, add 10 ml of ammonium molybdate solution, mix well and allow to stand for 15 minutes (pH of the solution at this stage is 1.0 to 1.2). Add while stirring 5 ml of oxalic acid solution and then 20 ml of sulphuric acid.

A-5.4.3 Transfer quantitatively to a 100-ml measuring flask. Mix, add 2 ml of the reducing solution, dilute to the mark and mix. Allow to stand for 20 minutes. Using the photoelectric absorptiometer fitted with suitable filter carry out photometric measurement using cells of optical path length 2 cm, after having adjusted the instrument to zero absorbance against water.

A-5.4.4 Carry out a blank test at the same time as the test portion using the same procedure and the same quantities of all reagents as in the case of test portion but omitting the sample.

A-5.4.5 Preparation of Calibration Graph

A-5.4.5.1 Into a series of six 100-ml measuring flasks, transfer 0, 1, 2, 4, 6 and 8 ml of the standard silica solution B.

A-5.4.5.2 Add in each flask 20 ml of water and while stirring 4 ml of dilute hydrochloric acid and 35 ml of boric acid solution. Allow to stand for 5 minutes, then add 10 ml of ammonium molybdate solution, mix and allow to stand for 15 minutes. Add, while stirring 5.0 ml of oxalic acid solution, 20 ml of dilute sulphuric acid and mix.

A-5.4.5.3 Add 2 ml of reducing solution, dilute to the mark and mix well. Allow to stand for 20 minutes and then carry out the photometric measurement exactly in the same manner as described above in case of the test portion. Deduct the absorbance reading of the blank test on the reagents from those of the standard silica solutions.

A-5.4.5.4 Plot a graph having the quantities in micrograms of hydrofluorosilicic acid (H_2SiF_6) in the standard silica solutions as abscissa and the corresponding absorbance values as the ordinates.

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A-5.4.5.5 Determine the quantities of hydrofluosilicic acid (H_2SiF_6) corresponding to the absorbance reading of the test solution and that of the blank test solution by means of the calibration graph.

A-5.5 Calculation

$$\text{Silica (as } \text{H}_2\text{SiF}_6 \text{), percent by mass} = \frac{(M_1 - M_2)}{100 M \times C}$$

where

M_1 = mass in g of the hydrofluosilicic acid in test solution,

M_2 = mass in g of the hydrofluosilicic acid in blank test solution,

M = mass in g of the test portion, and

C = concentration of anhydrous hydrogen fluoride expressed as percent by mass in the test sample (see **B-5.4**).

A-6. DETERMINATION OF PHOSPHORUS AS PHOSPHORUS OXIDE P_2O_5

A-6.1 Principle of the Method — Removal of fluorine and silica by evaporation of the test sample. Formation of phosphomolybdic acid complex in acidic solution. Reduction of the yellow-coloured complex to a blue-coloured molybdenum blue complex. Photometric measurement of the intensity of blue colour at about 610 nm wave length.

A-6.2 Reagents

A-6.2.1 Perchloric Acid — 60-70 percent.

A-6.2.2 Ammonium Molybdate Solution — Dissolve 10 g of ammonium molybdate tetra hydrate $[(\text{NH}_4)_6\text{Mo}_7\text{O}_{24}]$ in 100 ml of water. Add slowly 220 ml of sulphuric acid (1 : 1 solution) and 70 ml of water. Allow to cool and mix thoroughly.

A-6.2.3 Reducing Solution

A-6.2.3.1 Add 0.5 g of 1-amino-2-naphthol-4-sulfonic acid, 29.3 g of anhydrous sodium metabisulphite ($\text{Na}_2\text{S}_2\text{O}_5$) and 1 g of anhydrous sodium sulphite (Na_2SO_3) to 200 ml of water and warm on a water bath until complete dissolution. Filter and store in a cool dark place.

OR

Dissolve 0.25 g of 1-amino-2-naphthol-4-sulphonic acid and 0.5 g of anhydrous sodium sulphite in about 70 ml of water.

A-6.2.3.2 Dissolve 24 g of anhydrous sodium metabisulphite ($\text{Na}_2\text{S}_2\text{O}_5$) in about 100 ml of water.

A-6.2.3.3 Mix both the solutions and dilute to 200 ml with water. Filter, if necessary, and store in a cool dark place.

A-6.2.4 *Standard Phosphate Solution*

A-6.2.4.1 *Standard solution corresponding to 0.10 g of P_2O_5 per litre* — Weigh accurately 1.9175 g of pulverized dry potassium dihydrogen phosphate KH_2PO_4 , dissolve in water in a 1 000-ml measuring flask and dilute up to the mark. Mix thoroughly.

Transfer 10.0 ml of this solution to a 100-ml measuring flask, dilute to the mark and mix.

One millilitre of this solution corresponds to 0.1 mg (100 micro g) of P_2O_5 .

A-6.2.4.2 *Standard solution corresponding to 0.02 g of P_2O_5 per litre* — Transfer 50 ml of solution (**A-6.2.4.1**) to a 250-ml measuring flask, dilute to the mark and mix. One millilitre of this solution corresponds to 0.02 mg (20 micro g) of P_2O_5 .

A-6.3 **Apparatus**

A-6.3.1 *Platinum Dish* — 100 ml capacity.

A-6.3.2 *Photoelectric Absorption Meter* — Fitted with cells of 2 cm optical path and with filters of about 610 nm wave length.

A-6.4 **Procedure**

A-6.4.1 Weigh a quantity of test solution prepared in accordance with **B-5.3** containing not more than 0.5 g of hydrogen fluoride into a tared platinum dish, and weigh again. Add about 20 ml of water and evaporate the solution nearly to dryness on a waterbath.

A-6.4.2 Add 2 ml of perchloric acid and evaporate again nearly to dryness.

A-6.4.3 Add about 20 ml of water and transfer the mass quantitatively into a 100 ml measuring flask.

A-6.4.4 Add 10 ml of ammonium molybdate solution and 5 ml of reducing solution. Dilute with water to the mark. Place the flask in boiling water for 15 minutes and cool it with running water.

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A-6.4.5 Transfer the solution into the cell of 2 cm optical path length and carry out photometric measurement on photoelectric absorption meter after having adjusted the instrument on zero absorbance against water.

A-6.4.6 Carry out a blank test at the same time using the same procedure and the same quantities of all reagents as in the case of test sample but omitting the sample.

A-6.4.7 Preparation of calibration graph.

A-6.4.7.1 Transfer 1, 2, 4, 6, 8 and 10 ml of standard phosphate solution (A-6.2.4.2) into a series of six 100-ml measuring flasks.

A-6.4.7.2 Add 2 ml of perchloric acid, 20 ml of water and proceed as mentioned in A-6.4.4 and A-6.4.5, finally carrying out photometric measurements on the photometer, after adjusting the instrument on zero absorbance against water.

A-6.4.7.3 Plot a graph having the quantities in micrograms of phosphorous (P_2O_5) in the standard phosphate solutions as abscissa and the corresponding absorbance values as the ordinates.

A-6.5 Calculation — Determine the quantities of phosphorous (P_2O_5) corresponding to the absorbance reading of the test solution and that of the blank test solution by means of the calibration graph. The phosphorous content (P_2O_5) is given by the following formula:

$$\begin{array}{l} \text{Phosphorous (as } P_2O_5 \text{), percent} \\ \text{by mass} \end{array} = \frac{(M_1 - M_2)}{100 M \times C}$$

where

M_1 = mass in micrograms of phosphorous oxide (P_2O_5) in test solution,

M_2 = mass in micrograms of phosphorous oxide (P_2O_5) in blank test solution,

M = mass in g of the test sample solution, and

C = concentration of anhydrous hydrogenfluoride expressed as percent by mass in the test sample (see B-5.4).

A-7. DETERMINATION OF HYDROGEN FLUORIDE CONTENT

A-7.1 Calculation

Hydrogen fluoride
(as H F) content,

$$\text{percent by mass} = 100 - (\text{sum of all the impurities as determined in A-2, A-3, A-4, A-5 and A-6}).$$

APPENDIX B

(Clause 4.1)

SAMPLING OF ANHYDROUS HYDROGEN FLUORIDE, TECHNICAL

B-1. GENERAL REQUIREMENTS OF SAMPLING

B-1.0 In drawing samples, the following precautions and directions shall be observed.

B-1.1 Precautions shall be taken to protect the samples, the material being sampled, the sampling equipment and the containers for samples from adventitious contamination.

B-1.2 To draw a representative sample, the contents of each container shall be mixed thoroughly by rolling, shaking or stirring by suitable means and with necessary caution.

CAUTION: (a) Anhydrous hydrogen fluoride is a highly corrosive liquid, it attacks glass, has a great affinity for water and its vapour is irritant and toxic. Its action on the skin and eyes produces severe and painful burns which respond slowly to treatment.

(b) Samples shall be handled only inside a well ventilated fume-cupboard. Rubber gloves, boot and gown of suitable size to give adequate protection to the individual and full head and face protection should be used when handling.

(c) In case of contact or suspected contact, flood with sodium bicarbonate solution, then with water and seek immediate medical attention.

B-1.3 The samples shall be placed in suitable, clean, dry and air tight stainless steel containers.

B-1.4 Each sample container shall be sealed air-tight after filling and shall be marked with full details of sampling, the date of sampling and the year of manufacture of the material.

B-2. SCALE OF SAMPLING

B-2.1 Lot — All the containers in a single consignment of the material drawn from a single batch of manufacture shall constitute a lot. If a consignment is declared to consist of different batches of manufacture,

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the batches shall be marked separately and the groups of containers in each batch shall constitute separate lots.

B-2.2 Samples shall be tested from each lot separately for judging the conformity of the material to the requirements of the specification. The number of containers to be selected from lots of different sizes shall be in accordance with Table 2.

TABLE 2 NUMBER OF CONTAINERS TO BE SELECTED FOR SAMPLING

(*Clauses B-2.2 and B-2.3*)

LOT SIZE (<i>N</i>)	NO. OF CONTAINERS TO BE SELECTED (<i>n</i>)	LOT SIZE (<i>N</i>)	NO. OF CONTAINERS TO BE SELECTED (<i>n</i>)
(1)	(2)	(1)	(2)
Up to 15	2	101 to 300	6
16 to 25	3	301 to 500	7
26 to 50	4	501 to 1 000	8
51 to 100	5	1 001 and above	9

B-2.3 The containers shall be selected at random from the lot. In order to ensure the randomness of selection, procedure given in IS : 4905-1968* may be followed.

B-3. PREPARATION OF TEST SAMPLES

B-3.0 General — Allowing the liquid from the bulk container to flow into a cooled evacuated stainless steel cylinder of not less than one litre total capacity.

B-3.1 Apparatus — The apparatus for sampling is shown in Fig. 1.

B-3.1.1 Stainless Steel Cylinder (A) — One litre capacity.

B-3.1.2 Stainless Steel Coupling Tube (B) — equipped at either end with stainless steel unions threaded at one end to fit into valve *E* of the container holding the acid and at the other end to fit the screw thread of the outlet point *E* of the cylinder.

B-3.1.3 Stainless Steel Valve C — fitting to the screw thread of the outlet point *F* of the cylinder.

*Methods for random sampling.

B-3.1.4 Stainless Steel Tube D — connecting the valve to the extraction system.

B-3.1.5 Vacuum Pump — capable of producing rapidly a pressure of about 0.6 kPa inside the cylinder.

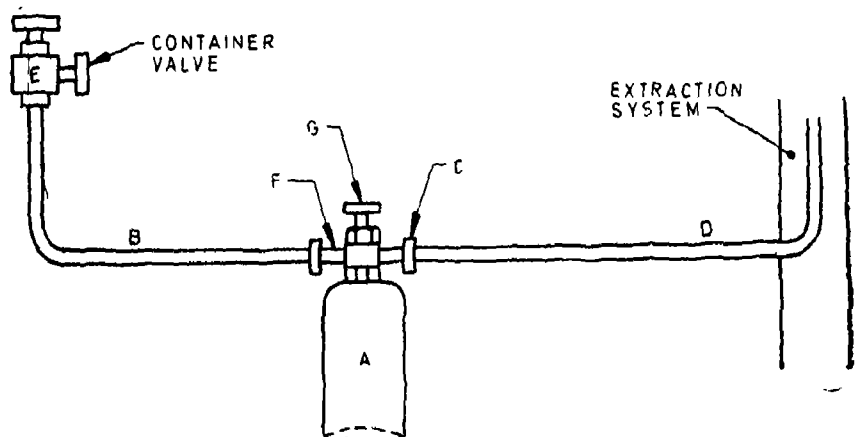


FIG. 1 EQUIPMENT FOR TAKING SAMPLES

B-3.2 Procedure

B-3.2.1 Cleaning and Drying

B-3.2.1.1 Connect the screw thread end *F* of the cylinder to the extraction system through valve *C* and the tube *D*. Open the cylinder valve to allow any residual hydrogen fluoride in the cylinder to be extracted. Close the valve of the cylinder after allowing sufficient time for complete extraction.

B-3.2.1.2 Disconnect the cylinder from the tube *D*, open valve *G* of the cylinder and wash thoroughly with water, allowing water to flow into the cylinder through the screw thread end.

B-3.2.1.3 Empty the cylinder and dry completely in an oven at 110°C. Store in the oven until required.

B-3.2.2 Evacuation — Evacuate the cylinder by means of the vacuum pump, close the valve and cool it to below 15°C.

B-3.2.3 Taking of Sample

B-3.2.3.1 Connect the tube *B* to the valve *E* of the container and to the valve *C* of the tube *D* leading to fume extraction system (see Fig. 2).

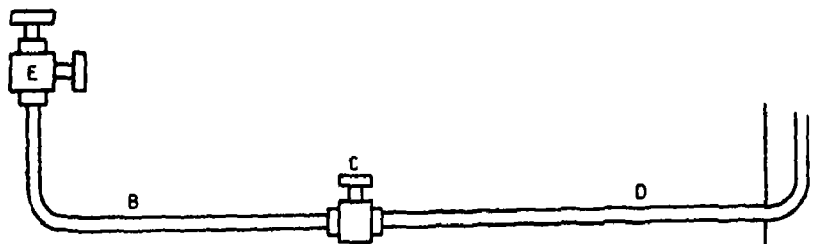


FIG. 2 EXTRACTION SYSTEM

B-3.2.3.2 Open the valve *E* and *C*, so that the sample will pass through the tube *B* and *D* to the extraction system, thereby purging the sampling line. Allow the purge to continue for 2 minutes.

B-3.2.3.3 Close the valve *E* and after about 2 minutes, disconnect that end of the tube *B*, which is connected to valve *C* and connect it immediately to the screw thread end *F* of the cylinder *A*.

B-3.2.3.4 Open valve *G* of the cylinder and then valve *E* of the acid container, allowing the sample to flow into the cylinder for about 3 minutes.

B-3.2.3.5 Close the valve *E* of the container and then the cylinder valve *G*.

B-3.2.3.6 Disconnect the cylinder from the coupling tube *B* very carefully allowing the little quantity of hydrogen fluoride left out in the tube *B* to drain out in a suitable polyvinyl chloride (PVC) container filled with ice pieces.

B-4. PREPARATION OF SAMPLE FOR DETERMINATION OF MOISTURE

B-4.0 General — Introduction of the liquid anhydrous hydrogen fluoride into preweighed dry polyethylene bottle through a cooling system.

B-4.1 Apparatus

B-4.1.1 Polyethylene Bottle with Extra Polyethylene Caps — three 250-ml capacity (see Fig. 3).

NOTE — The bottles should never be rinsed with water because polyethylene absorbs some water which is not easily removed and which slowly reacts with Karl Fischer reagent to give erroneous end point. Rinse the bottle with dry toluene.

B-4.1.2 Copper Cooling Coil — with flared connection at one end to fit cylinder valve adaptor and a flare union at the other end as shown in Fig. 3.

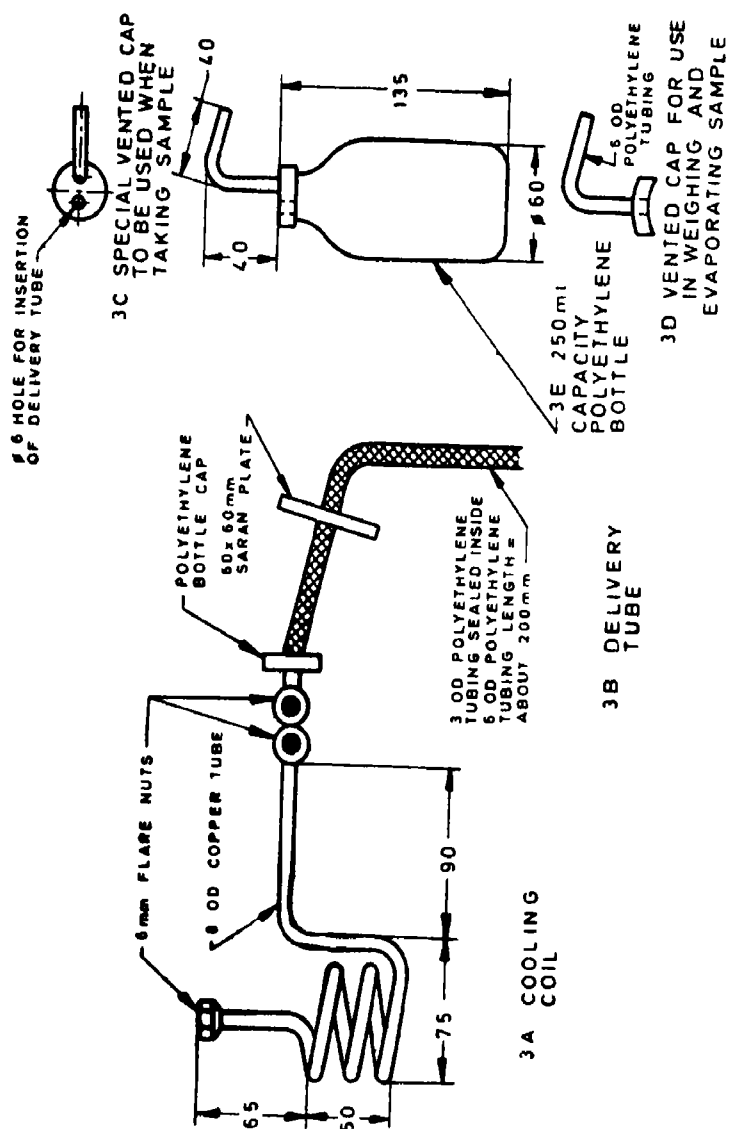


FIG. 3 EQUIPMENT FOR PREPARATION OF SAMPLE FOR DETERMINATION OF MOISTURE

B-4.1.3 Delivery Tube — made from 200 mm long piece of polyethylene tubing with 6 mm O.D. (the inner 3 mm O.D. sealed in polyethylene tube is optional). It is used in case of high humidity conditions to avoid condensate on the outside of the larger tube being introduced into the sample bottle.

B-4.1.4 Slip the polyethylene cap up to coupling and also slip on the saran plate as shown in Fig. 3. The arrangement blocks excessive condensate from trickling down the delivery tube and also covers the sampling bottle.

NOTE — Saran is a generic term for thermoplastic resin obtained by the polymerization of vinylidene chloride or co-polymerization of vinylidene chloride with lesser amounts of other unsaturated compounds.

B-4.1.5 Wooden Support — for sampling cylinder.

B-4.2 Procedure

B-4.2.1 Attach an adaptor with teflon washer to the valve outlet *F* of the cylinder and tighten it. Attach the copper coil to the adaptor and the polyethylene delivery tube to the flare union of the copper coil and tighten. Position the polyethylene beaker so that the copper coil remains inside the beaker and fill it with ice pieces.

B-4.2.2 Place a polyethylene waste container having some ice pieces under the delivery tube outlet and slowly open the valve of the sample cylinder *A*, to bleed off sufficient acid to purge the sampling line. Close the sampling valve, allow the delivery tube to drain out and then remove the waste container.

B-4.2.3 Dry the bent portion of the delivery tube outlet thoroughly with filter paper piece.

B-4.2.4 Weigh on the top pan balance, the sample bottle with vented cap. Replace the cap with a special-vented cap having a hole to receive the delivery tube as in Fig. 3 B.

B-4.2.5 Immediately after purging the line and wiping off any liquid condensing on the delivery tube, place the sample bottle at the outlet of the delivery tube, such that the end portion of the tube is inserted into the bottle through the hole in the vented cap. Place a polyethylene dish under the sample bottle and fill the dish with ice pieces. Allow a few minutes to cool the bottle below 20°C.

B-4.2.6 Open the cylinder valve and allow about 50 to 70 ml of the sample to run in rapidly. Loose the valve and allow the delivery tube to drain.

B-4.2.7 Remove the bottle with ice bath from the delivery tube, replace immediately the cap with the vented cap (*see* Fig. 3 D)

B-4.2.8 Remove the bottle from ice bath, wipe the outside dry and reweigh. Place the sample bottle on water bath (37.5°C) and allow the sample to evaporate.

NOTE — A small amount of fumes escaping from the vent tube, when the bottle is removed from ice bath, is negligible.

B-5. PREPARATION OF SAMPLE FOR OTHER DETERMINATIONS

B-5.0 General — Introduction of the liquid anhydrous hydrogen fluoride into a polyethylene cylinder containing the two layers of ice.

B-5.1 Apparatus — The apparatus is shown in Fig. 4.

B-5.1.1 Dilution Vessel H — of translucent polyethylene material of dia 60 mm and height 100 mm fitted with two lugs 120 mm from the bottom of the vessel.

B-5.1.2 Perforated Polyethylene Disc J — resting on the side lugs of the vessel H.

B-5.1.3 Polyethylene Stopper K — fitting on to the vessel H.

B-5.1.4 Delivery Tube L — of polyethylene material, 8 mm dia and 150 mm long.

B-5.1.5 Copper Connecting Tube M — 8 mm dia and 20 mm long fitting by means of threaded union to the cylinder valve adapter.

B-5.1.6 Polyethylene Connection Sleeve N — to join the connecting tube M to the delivery tube L.

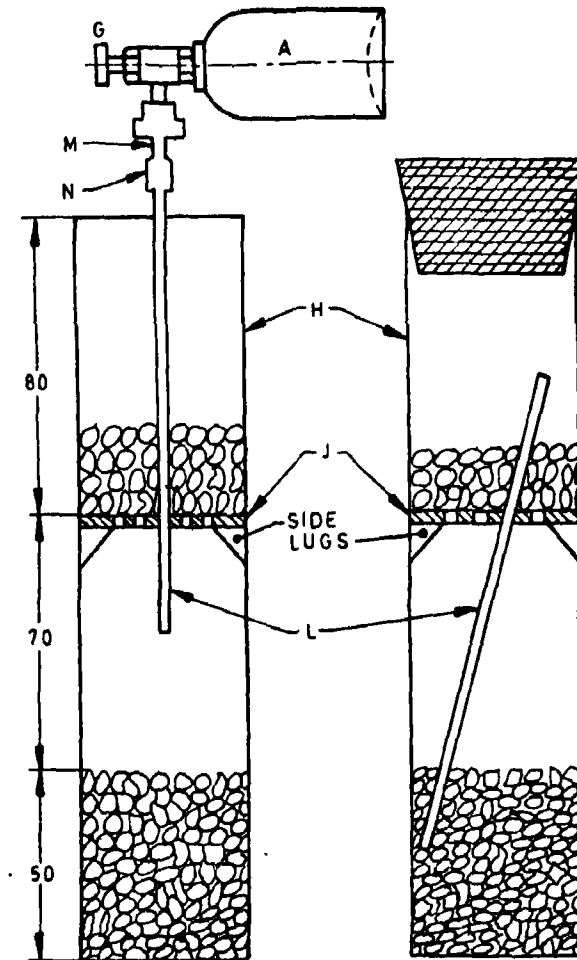
B-5.1.7 Wooden Support for Sample Cylinder

B-5.2 Reagents

B-5.2.1 Ice — prepared from distilled water.

B-5.3 Procedure

B-5.3.1 Insert the perforated disc J, delivery tube L and stopper K, to the previously dried dilution vessel. Weigh to the nearest 0.1 g. Remove the stopper, delivery tube and perforated disc.



All dimensions in millimetres.

FIG. 4 EQUIPMENT FOR PREPARING TEST SAMPLE FOR
OTHER DETERMINATIONS

B-5.3.2 Crush some ice to pieces of about 15 mm dia, rinse with water and introduce approximately 40 g into the dilution vessel *H*. Place perforated disc *J* on the side lugs of the vessel *H* and introduce delivery tube *L*, through the centre hole of the disc *J*. Add approximately 40 g of the ice pieces above the disc, wipe outside of the vessel with dry cloth to remove any condensed moisture and reweigh with stopper to nearest 0.1 g.

B-5.3.3 Remove the screw cap from the sample cylinder *A*, and connect the threaded union of the connecting tube *M* to the cylinder. Join the other end of the connecting tube to delivery tube *L*, by means of connection sleeve *N*.

B-5.3.4 Position to the bottom end of the delivery tube in the empty space below the perforated disc and carefully open the cylinder valve *G*. Allow anhydrous hydrogen fluoride to flow drop by drop until approximately 30 g are introduced.

B-5.3.5 Close the valve and disconnect the delivery tube, allowing it to drop into the dilution vessel *H*. Insert the stopper *K* to the vessel *H*. Disconnect the connecting tube *M* from the cylinder *A* and refit the screw can.

B-5.3.6 Wipe outside of the dilution vessel with a dry cloth to remove any condensed moisture and reweigh the vessel *H* to nearest 0.1 g. Agitate the dilution vessel until the ice melts completely and then transfer the contents to a 250-ml dry polyethylene bottle with screw cap.

B-5.4 Calculation

$$\text{Anhydrous hydrogen fluoride,} \\ \text{percent by mass} = \frac{M_1}{M_1 + M_2} \times 100$$

where

M_1 = mass in g of anhydrous hydrogen fluoride introduced,
and

M_2 = mass in g of ice used.

B-6. TEST SAMPLES AND REFEREE SAMPLE

B-6.1 From each of the containers selected according to B-2.2 a representative sample of the material shall be drawn according to procedure given in B-3.2.3. From samples representing different containers, a small but approximately equal quantity of the material shall be taken and thoroughly mixed to form a composite sample sufficient to carry out triplicate determination for all the characteristics specified. The composite samples so obtained shall be divided into three equal parts, one for the purchaser, another for the supplier and the third for the referee.

B-6.2 The samples so obtained shall be transferred to separate stainless steel containers. These containers shall then be sealed air-tight and labelled with full identification particulars given in B-1.4.

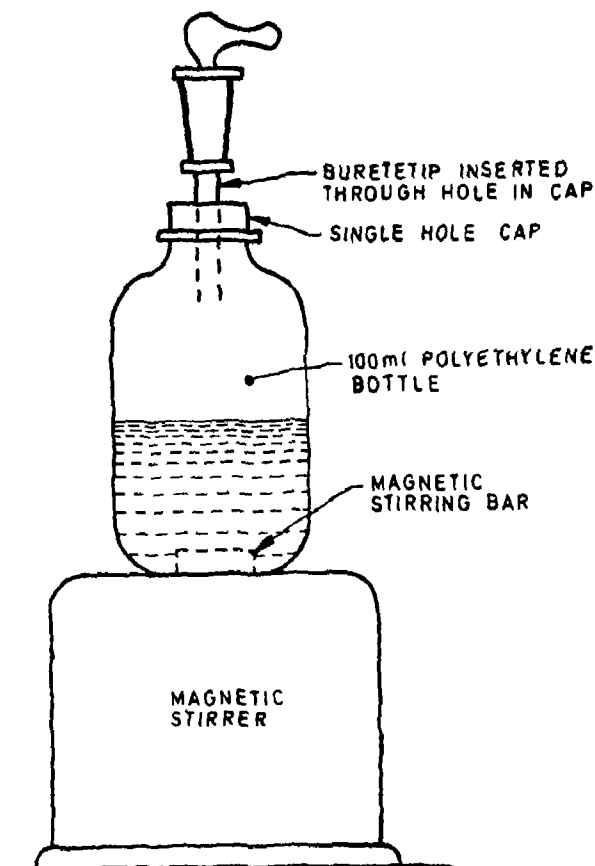


FIG. 5 EQUIPMENT FOR TITRATING WATER SAMPLE

B-6.3 The referee samples bearing the seals of both the purchaser and the supplier shall be kept at a place agreed to between the two. This shall be used in case of dispute between the two.

B-7. NUMBER OF TESTS AND CRITERIA FOR CONFORMITY

B-7.1 All the characteristics given under 2 shall be tested on the composite sample.

B-7.2 The lot shall be declared to have conforming to this specification if all the test results on the composite samples meet the corresponding specification requirements.

(Continued from page 2)

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INTERNATIONAL SYSTEM OF UNITS (SI UNITS)

Base Units

QUANTITY	UNIT	SYMBOL
Length	metre	m
Mass	kilogram	kg
Time	second	s
Electric current	ampere	A
Thermodynamic temperature	kelvin	K
Luminous intensity	candela	cd
Amount of substance	mole	mol

Supplementary Units

QUANTITY	UNIT	SYMBOL
Plane angle	radian	rad
Solid angle	steradian	sr

Derived Units

QUANTITY	UNIT	SYMBOL	DEFINITION
Force	newton	N	$1 \text{ N} = 1 \text{ kg.m/s}^2$
Energy	joule	J	$1 \text{ J} = 1 \text{ N.m}$
Power	watt	W	$1 \text{ W} = 1 \text{ J/s}$
Flux	weber	Wb	$1 \text{ Wb} = 1 \text{ V.s}$
Flux density	tesla	T	$1 \text{ T} = 1 \text{ Wb/m}^2$
Frequency	hertz	Hz	$1 \text{ Hz} = 1 \text{ c/s (s}^{-1}\text{)}$
Electric conductance	siemens	S	$1 \text{ S} = 1 \text{ A/V}$
Electromotive force	volt	V	$1 \text{ V} = 1 \text{ W/A}$
Pressure, stress	pascal	Pa	$1 \text{ Pa} = 1 \text{ N/m}^2$

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